

Characterization of Lubricants on Ball Bearings by FT–IR Using an Integrating Sphere

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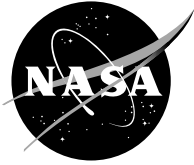
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Acknowledgments

This work was supported by the Revolutionary Aeropropulsion Concepts subproject of the Propulsion and Power Project at the NASA Glenn Research Center. The authors gratefully acknowledge Mario Marchetti for assistance with the spiral orbit tribometry samples and Duane Dixon for his assistance with fabricating sample holders.

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This work was sponsored by the Vehicle Systems Program at the NASA Glenn Research Center.

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Abstract

Fourier Transform-Infrared reflectance microspectroscopy has been used extensively for the examination of coatings on nonplanar surfaces such as ball bearings. While this technique offers considerable advantages, practical application has many drawbacks, some of which are easily overcome by the use of integrating sphere technology. This paper describes the use of an integrating sphere for the quantification of thin layers of lubricant on the surface of ball bearings and the parameters which require optimization in order to obtain reliable data. Several applications of the technique are discussed including determination of lubricant load on 12.7 mm steel ball bearings and the examination of degraded lubricant on post mortem specimens.

Introduction

Fourier transform infrared (FT-IR) spectroscopy has been used extensively over the years to characterize lubricants and their degradation products (refs. 1 to 6). The introduction of the microscope attachment to the FT-IR has greatly facilitated examination of the degradation products by allowing spectra to be acquired from selected areas of curved surfaces such as ball bearings and races. Typically, direct reflectance objectives are employed (refs. 1 to 7) although grazing angle objectives (refs. 5 and 8) have been used. When small amounts of material are present FT-IR microscopy can examine small particles of wear debris or aggregated materials. For surfaces covered with a homogeneous material having a constant thickness, the microscope can also provide quantitative estimates of film thickness. A general limitation of the technique in these instances is that the curved surface must be positioned such that the crown of the surface is centered in the objective in order to recover an adequate portion of the reflected light to obtain the desired spectrum.

A major limitation of infrared microspectroscopy arises where the coating thickness is not uniform over the surface and quantification is desired. Analysis thus requires spectral information from the entire surface or at least a statistically significant fraction thereof. However, to examine the surface of a 12.7 mm diameter ball bearing with a 0.1 mm diameter viewing objective, over 10^6 spectra would be required and this is not feasible.

Integration spheres have long been available as attachments for FT-IR spectrometers. Although they are used traditionally for scattering transmission or reflectance measurements from materials and surface coatings, it is also possible to mount objects having symmetrical geometry such as balls within integrating spheres and to obtain spectra from significant portions of the complete surface, thus averaging over local heterogeneity. In this paper we introduce the technique of using an integrating sphere for characterizing

lubricants and degradation products that are not distributed uniformly over the ball bearings surface. Quantification of a single substance and qualitative chemical characterization of a mixed substance will be addressed. Requirements for instrument modification, parameter optimization and other criteria necessary for obtaining quantitative information for lubricants on ball bearings will be presented as well as applications of the technique.

Experimental

Infrared spectra were collected with a “Magna 760” (Thermo Fisher Scientific, Inc., formerly Nicolet Instrument Corporation) FTIR spectrometer with a DTGS (deuterated triglycine sulfate) detector mounted within a 7.6 cm diameter gold coated integrating sphere (Labsphere, Inc.) accessory. The sample was mounted in the sphere on a gold plated holder located in the reflectance position. Two sample holders were used, one for 12.7 mm balls and one for less than 12.7 mm diameter balls. The 12.7 mm sample holder contained a 9.5 mm hole such that the ball mounted in the hole was centered in the light beam. For mounting balls smaller than 12.7 mm diameter, a similar solid aluminum plate was machined with a centered conical notch (6 mm diameter and 1 mm depth) which was then gold plated. Infrared microspectroscopy was performed with a NicPlan microscope (Spectra Tech, Inc.) equipped with a 32X reflectance objective and an MCT detector. Spectra were acquired in reflectance mode against a gold microscope slide as reference. Spectra were acquired between 400 (650 for the microscope) and at least 4000 cm^{-1} at 4 or 8 cm^{-1} resolution ratioed to spectra of the diffuse gold coating of the sphere by rotating the beam steering mirror to direct the incoming infrared radiation onto the surface of the sphere. Between 100 and 1000 scans were typically averaged and converted from transmittance to absorbance. The spectra were further corrected to remove water vapor absorption bands and baseline curvature.

All specimens were AISI 440C stainless steel grade 25 ball bearings. The balls were first rubbed with a alumina slurry and rinsed under running deionized water, followed by ultrasonification in deionized water. Drying was done with filtered nitrogen.

Two lubricants employed in vacuum applications were used. Solutions of 0.65 μg per μg Pennzane 2001A per μl hexane and 2 μg Krytox 143AB per μl Freon were prepared. A ball was held against a horizontally rotating shaft at one point (designated here as its north pole) and a measured volume of the lubricant solution was applied to the rotating surface from a gas tight syringe. After the solvent evaporated, the weight gained by the ball was determined by a microbalance with resolution $\pm 2 \mu\text{g}$. The smallest weight of lubricant that can be reliably quantified in this manner is in the 10 μg range.

Figure 1 shows a simplified schematic of the integration sphere with ball bearing sample. The intensity of the incoming beam is not uniform, but is a complex function of the spectrometer aperture and beam steering optics within the FT-IR bench. The beam directing mirror is not located directly over the center of the sample causing the beam to fall onto the sample at a small angle (on the order of 10°) from normal. For a flat sample at the reflectance port, the beam converges on the center of the sample, but for

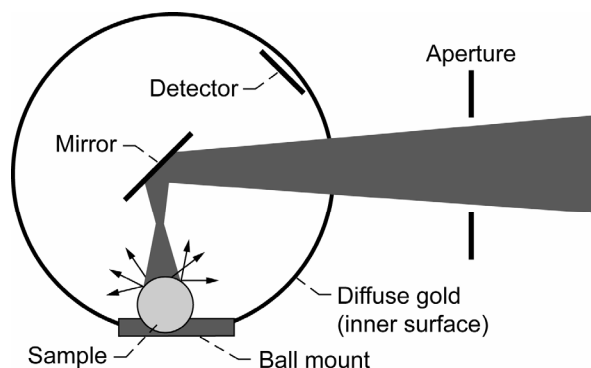


Figure 1.—Schematic of integrating sphere with sample.

three dimensional samples, the higher the sample, the further the center of the sample will be from the center of the beam. It is estimated that the beam covers between 20 and 50 percent of the surface of the ball (12.7 mm diameter) and depends primarily on (1) the aperture setting of the bench, (2) the ball diameter and (3) the positioning of the ball with respect to the center of the beam. Because of these uncertainties, it is believed that at least five spectra obtained from each ball are necessary to provide information from the entire surface area. The positions investigated on the ball included the top (north pole), bottom (south pole) and three evenly spaced positions about the equator (between top and bottom) of the ball. Care was taken to index all positions.

Samples of degraded lubricant were obtained by loading a 12.7 mm diameter ball charged with a mixture of Krytox 143AB and nano-onions into a spiral orbit tribometer and running to failure. Details on the spiral orbit Tribometer (refs. 9 and 10) and the Krytox/nano-onion experiment (ref. 11) are described in the literature.

Results and Discussion

Quantitative Aspects

In this section, quantification of the lubricant charge on balls lubricated as described above is provided. The lubricant coverage is not uniform as indicated by visually observed interference bands on the ball and also by the variation in signal intensity when examined in the infrared microscope. Figure 2 shows a representative set of infrared spectra of the C-H stretch bands for 50 μg of lubricant on a 12.7 mm diameter ball. This figure illustrates the variation in the thickness of Pennzane lubricant delivered to the ball by the syringe loading technique as measured by absorbance. There is no pattern to the deposition from one ball to the next even though when each ball is loaded it is held by the top and rotated about the equator. Figure 3(a) is a plot of the average absorbance for the 5 positions where the spectra were taken on different 12.7 mm balls versus the lubricant charge determined by the weight gain method. Figure 3(b) is a plot of the absorbance versus the lubricant charge expressed in the volume of solution delivered onto the spinning ball. It is clear that the plot where the oil is determined by weight gain has a lower linear correlation coefficient than the plot where the oil applied is related to the volume of oil standard delivered. This observation leads to the conclusion that the volumetric calculation of the amount of oil is at least as accurate as determination by weight gain, a time consuming process. Further, it is possible to reliably determine lower amounts of oil on the ball using spectrophotometry than by gravimetry. In figure 3(b), the test point, having approximately 6.5 μg lubricant, estimated by volumetric calculation, falls on the extrapolated curve prepared by using the data above 20 μl . In passing, these curves should intersect the absorbance axis at zero for the blank. The negative offset is most probably due

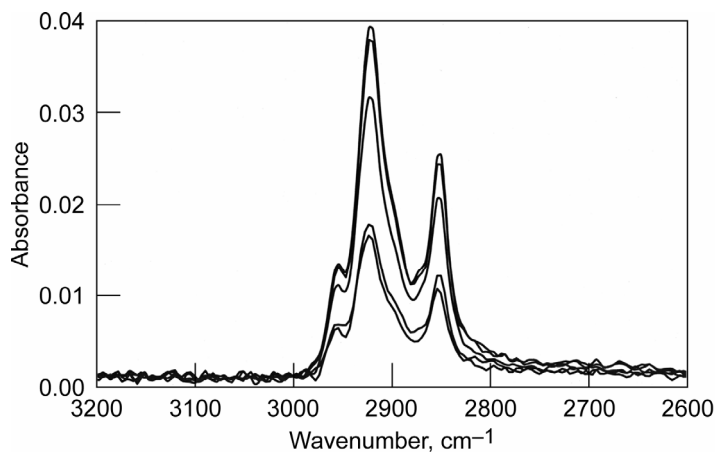


Figure 2.—Infrared spectra of the five ball positions for 50 μg of lubricant on a 12.7 mm diameter ball

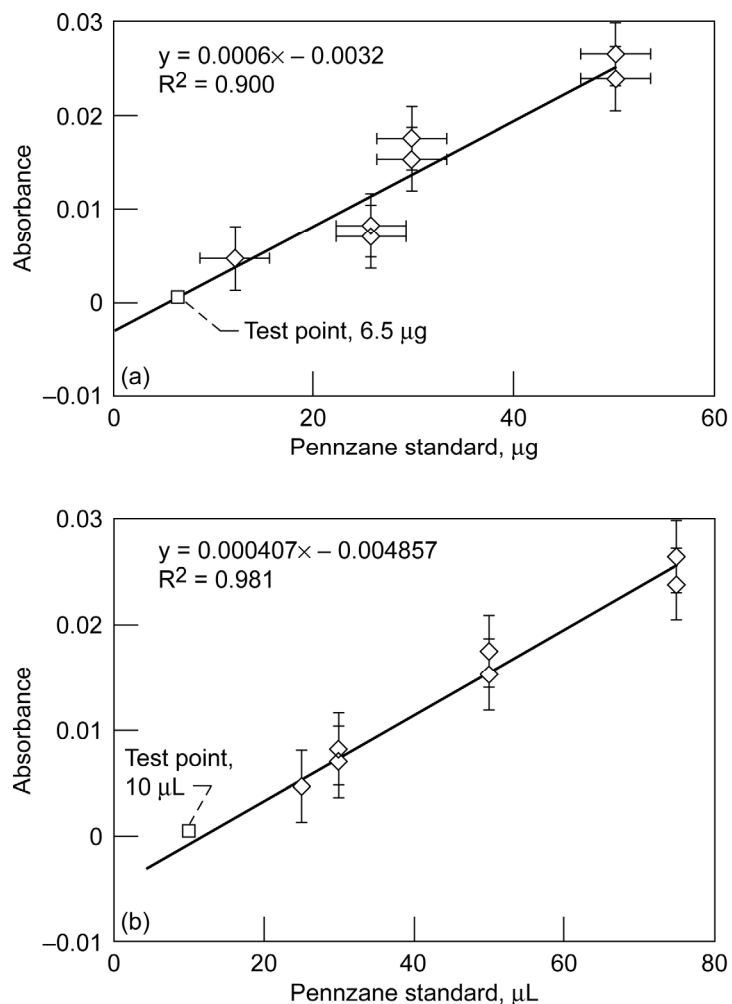


Figure 3.—Plot of the absorbance on 12.7 mm balls versus the lubricant charge. (a) Determined by the weight grain method. (b) Expressed as volume of solution delivered.

to a constant bias encountered when using the application software to determine the baseline corrected absorbance for the plots. The $6.5 \mu\text{g}$ spectra do in fact have positive peaks which are readily recognized from the noise in the baseline. The limit of detection, LOD, for Pennzane on 12.7 mm balls using the 2923 cm^{-1} peak in the infrared spectrum for detection is $2.0 \mu\text{g}$ based on three times the standard deviation in the noise on either side of the peak of the $12 \mu\text{g}$ standard spectra. It is possible that the LOD could be lowered by noise reduction techniques such as averaging more scans to acquire the spectra, scanning more slowly, or by mathematical manipulations of the spectra such as Savitsky-Golay or FT smoothing. For example, when the spectra for all 5 evaluated positions on the ball are averaged and those spectra for duplicate determinations are averaged, the LOD is lowered to $0.9 \mu\text{g}$.

For the data in figure 3, all spectra except one for $50 \mu\text{g}$ were the average of 1000 scans. The other $50 \mu\text{g}$ spectrum was the average of 250 scans. For higher concentrations of lubricant on the ball it is possible to obtain reliable information from fewer scans, but not so for lower amounts of lubricant where the spectral features become buried in the noise. It is cautioned that even small lubricant loadings migrate in time such that spectra used for quantitative purposes should be collected in as short of time as possible. For this reason, no more than 1000 scans were averaged to obtain individual spectra for quantification. All quantitative spectra used in this work were run at 8 cm^{-1} resolution to expedite collection time which

is a function of spectral resolution. At 8 cm^{-1} , the spectra maintain most of the spectral features necessary to characterize the lubricant, whereas the spectra collected at 16 cm^{-1} are significantly degraded for qualitative purposes.

Parameter Optimization

Aside from the aforementioned effects of spectral resolution and number of scans on acquiring reliable absorbance information for quantification, several other parameters need to be considered, including aperture setting and specimen positioning, which are interrelated. Opening the aperture increases the beam diameter effectively letting more light to the detector which improves signal to noise ratio. Opening the aperture too much allows light energy to spill over the edge of the ball onto the sample holder resulting in a fraction of the radiation not impinging on the sample prior to being measured by the detector. A similar effect is observed if the ball is not centered in the beam. The optimum aperture setting is thus a function of the size of the ball under investigation. Figure 4 shows the effect of aperture setting on absorbance for 12.7, 9.53 and 6.35 mm balls loaded with 206 ($0.406\text{ }\mu\text{g}/\text{mm}^2$), 116 ($0.407\text{ }\mu\text{g}/\text{mm}^2$) and 58 ($0.45\text{ }\mu\text{g}/\text{mm}^2$) μg Pennzane respectively. To account for small changes in surface coverage, the absorbance data was normalized to the aperture setting of 100. Figure 4 shows the data for all balls mounted on the plate with centered conical hole described in the experimental section. In theory, the absorbance as defined by Beer's law should be independent of the aperture opening. In practice, the opening of the aperture lets more light fall on the edges of the ball where the pathlength experienced by the peripheral infrared energy strikes the ball at oblique angles leading to higher absorbance values. Eventually, the aperture becomes large enough that significant amounts of light fall outside the circumference of the ball leading to negative slopes. Providing the slope is only slightly negative, as in the 12.7 and 9.53 mm balls, there is still advantage to be gained by opening the aperture which decreases the noise level in the spectra. For the 6.35 mm ball, there will be an optimum aperture setting where signal-to-noise ratio is maximized. With better positioning and aperture optimization it should be possible to do quantitative work with balls as small as 6.35 mm. It should also be possible to examine even smaller balls with a suitable beam condenser placed in front of the entrance to the integrating sphere.

Additional benefit is derived by the incident infrared beam striking the ball at oblique angles. At angles other than normal incidence, the beam traverses a greater pathlength through the sample layer on the ball, and at high angles simulates the enhancements observed in grazing angle spectrophotometry. Calculations using the optical constants in the literature (ref. 12) for Pennzane 2001 indicate a greatly

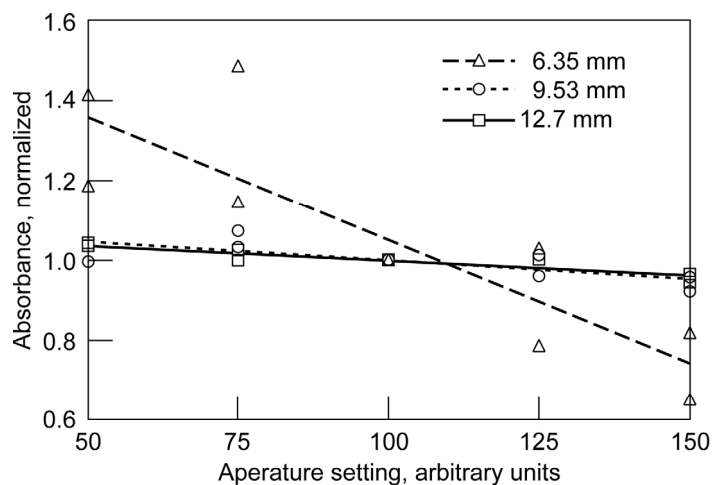


Figure 4.—Effect of aperture setting on normalized absorbance for 6.35, 9.53 and 12.7 mm balls loaded with a constant thickness of Pennzane.

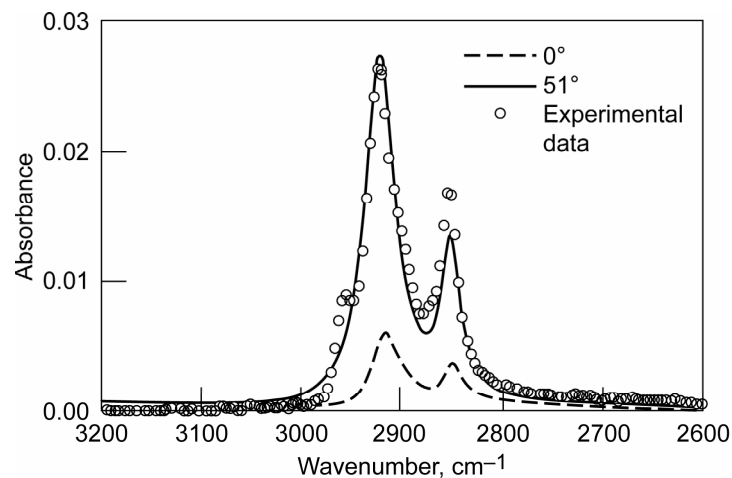


Figure 5.—Model of spectrum at beam angles of incidence 0° and 51° compared to experimental data for 50 μg Pennzane on 12.7 mm ball.

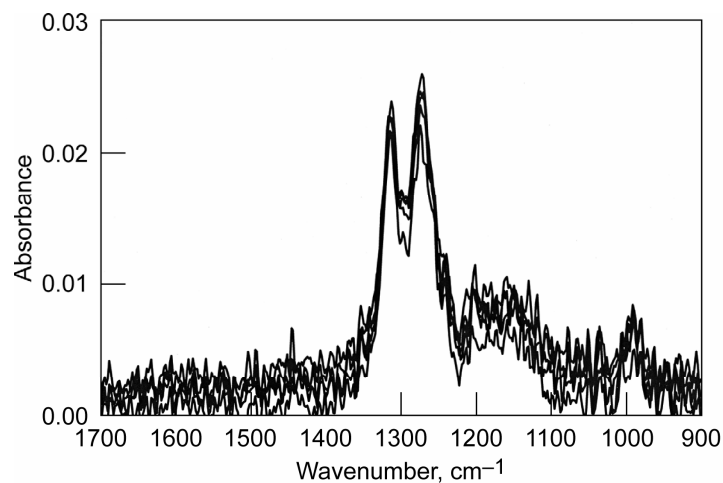


Figure 6.—Distribution of material on a ball after lubricant failure as determined by infrared spectra from five positions on the ball.

enhanced absorbance in the actual spectra over what would be predicted using two passes through the sample on the ball at normal incidence as seen in figure 5. In the current configuration a five fold enhancement is observed. Modeling of the spectrum indicates that the average angle of beam incidence on sample film on the order of 51° is required to produce model spectra similar in intensity to the actual spectra. The model spectra only include the CH_2 asymmetric absorption band at 2922 cm^{-1} and the symmetric band at 2851 cm^{-1} . The asymmetric CH_3 band at 2955 cm^{-1} is not used in the model (ref. 13).

Qualitative Aspects

It is also possible to obtain qualitative information using the technique. Figure 6 illustrates the distribution of material on a ball after lubricant failure in the tribometer (approximately 180,000 orbits) (ref. 11). The lubricant which was not so evenly distributed by syringe application has now distributed itself evenly over the ball such that the five spectra are identical to within experimental error, thus, any spectrum presents a global representation of the ball. This is not the case using the microscope where

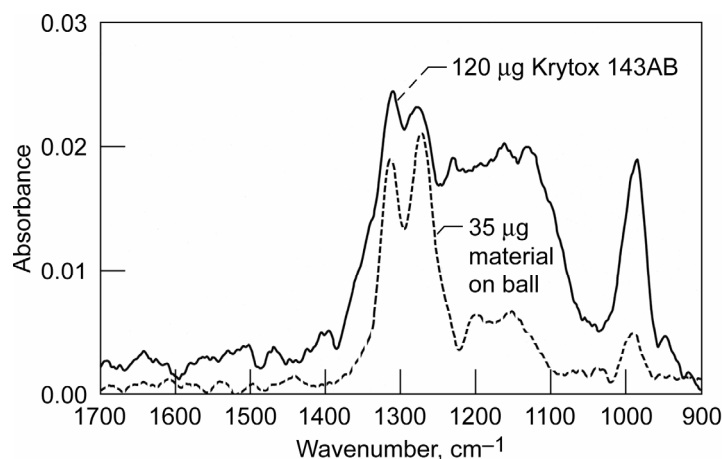


Figure 7.— Spectra of Krytox 143AB degradation product and Krytox 143AB standard on a 12.7 mm ball.

experience has shown that on the microscopic scale, there is considerable variation in the spectra from spot to spot, especially where there is little lubricant left. These spectra represent 35 µg of Krytox 143AB degradation product on the ball. This much Krytox 143 AB would behave as a lubricant with a friction coefficient of around 0.13, whereas this ball at failure had a friction coefficient of at least 0.28, the default value for experiment termination. Since the material washed freely from the ball leaving no traces of C-F stretch bands in the resulting spectrum, none of the 35 µg of material was polymerized. Hence it is concluded that the degraded material is a fluorocarbon similar to the original lubricant which has a similar spectrum, shown in figure 7, but is of significantly lowered molecular weight such that it would not serve as a lubricant.

Financial Considerations

Most modern spectrometers are capable of directly incorporating accessories such as integrating spheres. Some are not accommodating of accessories such as a microscope. Thus it is easier to implement the integrating sphere technique into an existing bench as the spheres are considerably less expensive (by about 5 to 10 times) than the microscopes with various objectives. The trade-off between these choices is that quantification is best performed with the proposed integrating sphere method but for characterization of small features, the microscope technique must be employed.

Conclusions

- 1) It is possible to use the integration sphere technique for quantitative analysis of lubricants on ball bearings by using suitable reference standards, which is not obtainable by FT-IR microscopy and which has a lower limit of detection than weighing.
- 2) It is possible to obtain quantitative information from a variety of ball sizes however; numerous parameters, especially aperture settings, and sampling geometry must be optimized.
- 3) Useful qualitative information is easily obtainable on unknown materials using this technique as a single spectrum examines a large fraction of the surface of a ball which might not be homogeneously covered.
- 4) The integration sphere technique provides complementary information to FT-IR microspectroscopy as it allows averaging over much greater areas, whereas the microscope is especially good for looking at small features.

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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE January 2007		3. REPORT TYPE AND DATES COVERED Technical Memorandum
4. TITLE AND SUBTITLE Characterization of Lubricants on Ball Bearings by FT-IR Using an Integrating Sphere			5. FUNDING NUMBERS WBS-22-066-30-04	
6. AUTHOR(S) K.W. Street, S.V. Pepper, A.A. Wright, and B. Grady				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration John H. Glenn Research Center at Lewis Field Cleveland, Ohio 44135-3191			8. PERFORMING ORGANIZATION REPORT NUMBER E-15764	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001			10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-2007-214472	
11. SUPPLEMENTARY NOTES K.W. Street and S.V. Pepper, NASA Glenn Research Center, Cleveland, Ohio; A.A. Wright, Spelman College, 350 Spelman Lane, Atlanta, Georgia 30314; and B. Grady, Ohio University, 1 Ohio University, Athens, Ohio 45701. Responsible person, K.W. Street, organization code RST, 216-433-5032.				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Category: 25 Available electronically at http://gltrs.grc.nasa.gov This publication is available from the NASA Center for AeroSpace Information, 301-621-0390.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Fourier Transform-Infrared reflectance microspectroscopy has been used extensively for the examination of coatings on nonplanar surfaces such as ball bearings. While this technique offers considerable advantages, practical application has many drawbacks, some of which are easily overcome by the use of integrating sphere technology. This paper describes the use of an integrating sphere for the quantification of thin layers of lubricant on the surface of ball bearings and the parameters which require optimization in order to obtain reliable data. Several applications of the technique are discussed including determination of lubricant load on 12.7 mm steel ball bearings and the examination of degraded lubricant on post mortem specimens.				
14. SUBJECT TERMS Spectroscopy			15. NUMBER OF PAGES 14	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	

